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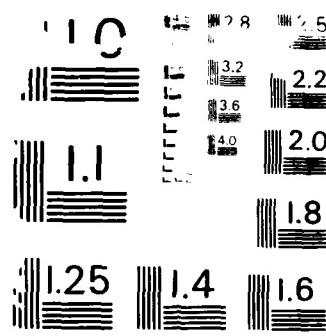
EFFECTS OF CADMIUM ELECTRODE PROPERTIES ON
NICKEL-CADMIUM CELL PERFORMANCE (U) AEROSPACE CORP EL
SEGUNDO CA CHEMISTRY AND PHYSICS LAB A H ZINNEMAN

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Effects of Cadmium Electrode Properties on Nickel-Cadmium Cell Performance

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30 December 1987

Prepared for
SPACE DIVISION
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This report was submitted by The Aerospace Corporation, El Segundo, CA 90245, under Contract No. F04701-85-C-0086 with the Space Division, P.O. Box 92960, Worldway Postal Center, Los Angeles, CA 90009-2960. It was reviewed and approved for The Aerospace Corporation by S. Feuerstein, Director, Chemistry and Physics Laboratory.

Lt Thomas Wetterstroem/CWASB was the project officer for the Mission-Oriented Investigation and Experimentation (MOIE) Program.

This report has been reviewed by the Public Affairs Office (PAS) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

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SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

1a REPORT SECURITY CLASSIFICATION Unclassified		1b. RESTRICTIVE MARKINGS	
2a SECURITY CLASSIFICATION AUTHORITY		3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited.	
2b DECLASSIFICATION/DOWNGRADING SCHEDULE			
4 PERFORMING ORGANIZATION REPORT NUMBER(S) TR-0086(6945-01)-2		5. MONITORING ORGANIZATION REPORT NUMBER(S) SD-TR-88-21	
6a. NAME OF PERFORMING ORGANIZATION The Aerospace Corporation Laboratory Operations	6b OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION Space Division Air Force Systems Command	
6c. ADDRESS (City, State, and ZIP Code) 2350 E. El Segundo Blvd. El Segundo, CA 90245		7b ADDRESS (City, State, and ZIP Code) Los Angeles Air Force Station P. O. Box 92960, Worldway Postal Center Los Angeles, CA 90009-2960	
8a. NAME OF FUNDING/SPONSORING ORGANIZATION	8b OFFICE SYMBOL (If applicable)	9 PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER	
8c. ADDRESS (City, State, and ZIP Code)		10 SOURCE OF FUNDING NUMBERS	
		PROGRAM ELEMENT NO	PROJECT NO.
		TASK NO	WORK UNIT ACCESSION NO.
11. TITLE (Include Security Classification) Effects of Cadmium Electrode Properties on Nickel-Cadmium Cell Performance			
12 PERSONAL AUTHOR(S) A. H. Zimmerman			
13a TYPE OF REPORT	13b TIME COVERED FROM _____ TO _____	14. DATE OF REPORT (Year, Month, Day) 1987 December 30	
15 PAGE COUNT 20			
16. SUPPLEMENTARY NOTATION			
17 COSATI CODES		18 SUBJECT TERMS (Continue on reverse if necessary and identify by block number)	
FIELD	GROUP	SUB-GROUP	
19 ABSTRACT (Continue on reverse if necessary and identify by block number)			
<p>Tests have been conducted on several nickel-cadmium cells that have exhibited a variety of performance problems, ranging from high voltages and pressures during overcharge to low capacity. The performance problems that have been specifically linked to the cadmium electrode are primarily related to two areas, poor sinter and the buildup of excessive pressure during overcharge. A number of specific nickel-cadmium cell and cadmium electrode characteristics have been studied in this work to determine what the effects of poor sinter are, and to determine what factors are important in causing excessive pressures during overcharge in cells that otherwise appear normal. Several of the tests appear suitable for screening cells and electrodes for such problems.</p>			
20 DISTRIBUTION/AVAILABILITY OF ABSTRACT <input type="checkbox"/> UNCLASSIFIED/UNLIMITED <input checked="" type="checkbox"/> SAME AS RPT <input type="checkbox"/> DTIC USERS		21. ABSTRACT SECURITY CLASSIFICATION Unclassified	
22a NAME OF RESPONSIBLE INDIVIDUAL		22b TELEPHONE (Include Area Code)	22c OFFICE SYMBOL

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I. BACKGROUND

In recent years significant variability between different production lots of nickel-cadmium cells has been a source of increasing concern among nickel-cadmium battery users. This report describes part of a study undertaken to address some of the main performance concerns, which include cells exhibiting high overcharge pressures and voltages as well as cells with low capacities. The objectives of this study are: (1) to determine the likely causes of performance variability and problems, and (2) to develop test methods capable of nondestructively detecting various incipient problems in NiCd cells early in life. The work reported here focuses on the cadmium electrode. The overall study also addresses the role of the nickel electrode and various cell contaminants in affecting performance, work that will be reported elsewhere.

To carry out this study, a number of nickel-cadmium cells exhibiting various performance problems were obtained along with nickel and cadmium electrodes from many of these cell lots. In addition, a large number of both nickel and cadmium electrode samples were obtained, some of which were associated with problem cell lots, and some of which were associated with normal NiCd cell lots. Because of recent concern over the effects of weak sinter on electrode performance, some cadmium electrode samples that were known to have very weak sinter were also included in this study. The performance problems that have specifically been linked to the characteristics of the cadmium electrode are primarily related to two areas, weak sinter and excessive pressures during overcharge. Both NiCd cell and cadmium electrode characteristics have been studied in this work to determine what the detailed effects of weak sinter are, and to determine what factors are important in causing excessive pressures during overcharge in cells that appear otherwise normal.

II. NICKEL-CADMIUM CELL TESTS

Testing on NiCd cells generally focused on problems with excessive overcharge pressures, since this situation is specific to internal cell environmental factors such as electrolyte distribution and oxygen transport rates that cannot be realistically simulated outside the actual NiCd cell. Tests were done on four different sizes of NiCd cells, which were 6, 10, 35, and 50 ampere hour (Ah) capacity. The 6 Ah cells were considered normal, i.e., representative of typical NiCd cell performance characteristics acceptable for space NiCd battery applications. The 10 and 35 Ah cells exhibited high internal pressures during 0°C overcharge at the C/20 rate (the C rate is the rate that will discharge the rated cell capacity in 1 hr). The 50 Ah cells exhibited very high voltages during overcharge. Voltages in excess of 1.57 volts were observed for one cell during 0°C overcharge at a C/20 rate.

Each cell was tested following the test sequence outlined in Table 1. The cells were tested in a chamber at a temperature that was regulated to 0 or 23 +1°C, depending on the portion of the test that was being run. Each cell was instrumented with a strain gauge to monitor pressure. However, the cases of some of the cells deformed sufficiently to cause debonding of the strain gauges, which prevented reliable pressure comparisons. Cell voltages were monitored continuously by a microprocessor controller with time resolution up to 1 ms.

The test outlined in Table 1 is designed to address a number of cell operating characteristics, and were evaluated for the purpose of developing some straightforward diagnostic signatures for several NiCd cell problem areas. Step 2 measures the voltage response of the cell to cyclic oxygen generation and recombination to determine if such recombination is significantly changing the electrode characteristics. The periodic steps in the charge current at various points in the test sequence are to measure the voltage response or impedance of the cell during various charge conditions.

Table 1. NiCd Cell Test Sequence

23°C Portion of Test

1. C/20 charge 40 hr, C/2 discharge to 1.0 volts, 1 ohm shortdown to make up 48 hr since start of step.
2. C/15 charge for 30 hr, 5 cycles consisting of 1 hr open circuit and 1 hr of C/15 charge. Store voltage at end of each charge segment.
3. C/2 discharge to 1.0 volt, 1 ohm shortdown to make 48 hr since start of step 2.
4. C/10 charge for 24 hr. Every 2 hr during this charge increase the current 20% (to C/8.33) for 400 sec and monitor the voltage response with 1 ms resolution.
5. Charge at C/20 for 800 sec, then at C/16.67 for 400 sec while monitoring the voltage with 1 msec time resolution.
6. Charge at C/50 for 1600 sec, then at C/41.67 for 800 sec while monitoring the voltage with 1 msec time resolution.
7. Charge at C/100 for 3200 sec, then at C/83.33 for 1600 sec while monitoring the voltage with 1 msec time resolution.
8. Charge at C/10 for 400 sec, then at C/8.33 for 200 sec while monitoring the voltage with 1 msec time resolution.
9. Charge at C/5 for 200 sec, then at C/4.167 for 100 sec while monitoring the voltage with 1 msec time resolution.
10. Charge at C/2.53 for 100 sec, then at C/3.29 for 50 sec while monitoring the voltage with 1 msec time resolution.
11. Discharge at C/2 to 1.0 volts, C/20 to 0.5 volts, then short with a 1 ohm resistor until 24 hr elapsed since start of step 5.

0°C Portion of Test

12. After stabilizing the temperature at 0°C, charge at C/20 for 48 hr. Every 2 hr during this charge increase the current to C/16.67 for 400 sec while monitoring the cell voltage with 1 msec time resolution.
13. Repeat steps 5-11.

The different rates of charge used in steps 5 to 10 are to try to establish an I/V curve during overcharge. To prevent overpressure at the higher charge rates in steps 5 to 10, low rates were initially used to allow the pressure to decrease to levels where higher charge rates could be briefly tolerated. I/V measurements in the cells met with only limited success because the cell voltage is sensitive to the oxygen pressure in the cell as well as the state of the nickel and cadmium electrodes.

Two 50 Ah NiCd cells were tested. Both of these 50 Ah cells were from a cell lot that had consistently developed quite high overcharge voltages during low temperature charging, behavior that was not present during acceptance testing by the manufacturer. The first of these cells, Cell 1, exhibited significantly higher overcharge voltages than did the second cell, Cell 2. At 0°C, Cell 1 developed such high voltages that the tests outlined in Table 1 could not be completed, but were automatically terminated by the safety features of the controlling system. The problems in these cells have been attributed to extreme loss of overcharge protection as a result of physical movement of cadmium active material in the cells. This problem is attributed to weak sinter in the cadmium electrodes, which is unable to effectively contain the active material. This scenario, which has been developed by the cell manufacturer, is consistent with the high voltages seen in Cell 1 in these tests during the overcharge condition.

A number of cells that had overcharge pressure problems were also studied, again to develop some approaches to nondestructively detecting these kinds of pressure problems based on cell performance characteristics. Cells 3 and 4 were from a 10 Ah cell lot that had developed moderately high pressures (enough to see some permanent deformation of the case tops, about 100 psi) during 0°C charging at C/20. Cell 3 had about 10% less electrolyte than did Cell 4. Cell 5 was a 10 Ah cell from a lot that had experienced significant cell case top bulging, with internal pressures estimated to have approached 150 psi during 72 hr of 0°C charging at C/20. Cell 6 was a 6 Ah cell that appeared to have normal operating characteristics and was used as a control in these studies.

The test in step 2 of Table 1 was done to accentuate differences arising from oxygen recombination in the NiCd cells by alternately generating oxygen and allowing it to recombine during open circuit stand. This was done using relatively benign conditions (C/15 charge at 23°C) so that a test of this type could be considered as not leading to any cell degradation, and thus could be generally applied to screening cells. The increases in cell voltage that occurred during the cyclic overcharge and open circuit periods of test step 2 are indicated in Fig. 1. The magnitude of the voltage increase in Fig. 1 gave a good correlation with the cells that exhibited performance problems, both high overcharge voltages and pressures. Cell 2, although from a lot of 50 Ah cells that tended to give high voltages during low temperature overcharge, did not produce an extremely high voltage. The effect of cell cycling on the behavior of Fig. 1 was also examined. Each cell was cycled 50 times (6 hr cycles, 50% depth of discharge, 102% charge return), after which they were retested. The cells having pressure problems tended to temporarily have slightly lower pressures after the cycling. Also, as indicated in Fig. 1 for cell 5, the cells had a reduced voltage rise during cyclic oxygen generation and recombination. It is also interesting to note that Cell 3, which had about 10% less electrolyte than did Cell 4 but was from the same lot, had about 4 mV less voltage rise in the test of Fig. 1. This indicates that while changing the amount of electrolyte can influence these characteristics, the overall signature is not extremely sensitive to significant variations in the amount of electrolyte.

The voltage increases of Fig. 1 range from 8 mV for a "normal" cell (Cell 6), to 10 mV for a cell that had slightly high overcharge voltages (Cell 2), to 16-19 mV for cells that were known to develop high pressures or high voltages during overcharge at 0°C. From these data alone it appears that an upward swing in voltage during the test in step 2 of 10 mV or less is desirable for properly operating cells. Cells that exhibit upward voltage swings significantly in excess of 10 mV appear to be candidates for problems with high voltages and/or pressures during low temperature overcharge. The differences in voltage swing for the cells indicated in Fig. 1 are expected to arise from changes in the cadmium electrode in response to oxygen recom-

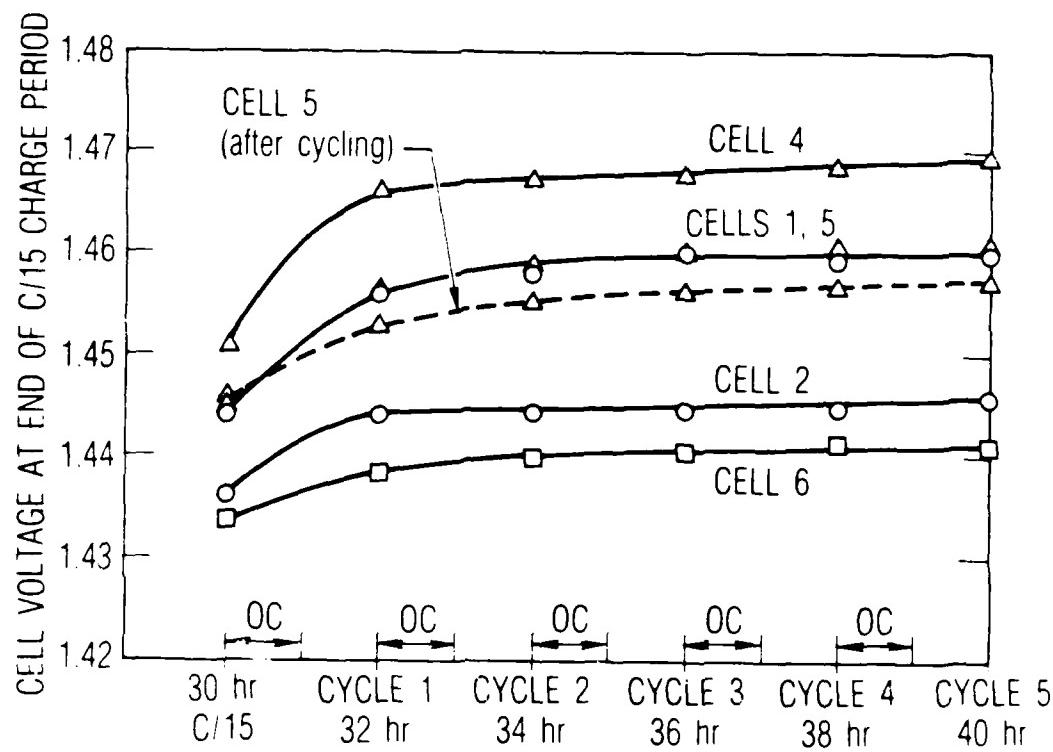


Fig. 1. Charge Voltage for NiCd Cells at Various Times During Intermittent C/15 Overcharge. Cell 6 is the 6 Ah control cell, Cell 2 a 50 Ah cell having slightly high overcharge voltages, Cell 1 a 50 Ah cell having extremely high overcharge voltages, and Cells 4 and 5 were 10 Ah cells having high internal pressures during overcharge at 0°C.

bination. While nickel electrode voltages could be affected by the intermittent open circuit periods in this test, the changes arising from the nickel electrode should be similar for all the cells tested. Thus, the correlation of the magnitude of the voltage increases in Fig. 1 with cell performance indicates the sensitivity of the cadmium electrode performance to oxygen evolution. This sensitivity must be kept below a critical level if the cadmium electrode is expected to operate in a stable fashion during overcharge over a wide range of temperatures and currents.

The other tests that were done as part of the sequence in Table 1 gave a systematic trend for the various problem cells to have slightly higher, or in the case of Cell 1, significantly higher voltages. The only other characteristic that was consistently found to correlate with cell problems associated with the cadmium electrode was the cell impedance during charge at 0°C at a C 20 rate, but prior to overcharge. When the cell approaches overcharge the voltage and the impedance become so strongly affected by the oxygen voltage at the nickel electrode that it is difficult to attribute any changes unambiguously to the cadmium electrode. However, prior to overcharge the impedance of the cadmium electrode can easily be resolved in either the time or frequency domain from that of the positive electrode. The cadmium electrode exhibits a rapid time response (~ 2-4 sec), whereas the nickel electrode has a very slow time response (>10 sec).

The voltage response to a small current step is directly proportional to the cell impedance and is indicated in Fig. 2 at the 8 hr point in a 48 hr C/20 charge at 0°C. The response within the first 4 sec is almost entirely due to the cadmium electrode. The cell IR drop, which gives an instantaneous voltage change, has been removed from the data of Fig. 2. The much slower voltage change occurring out to 250 sec is the response from the nickel electrode. The impedance of the cadmium electrode is expected to be strongly influenced by the electrolyte distribution in the cell during recharge as well as the ionic diffusion rates to the active electrode surface. It is interesting to note that only the cells that tended to develop high pressures during overcharge differ significantly in voltage response from the typical or "normal" cell behavior. The behavior in Fig. 2, therefore, may serve as a

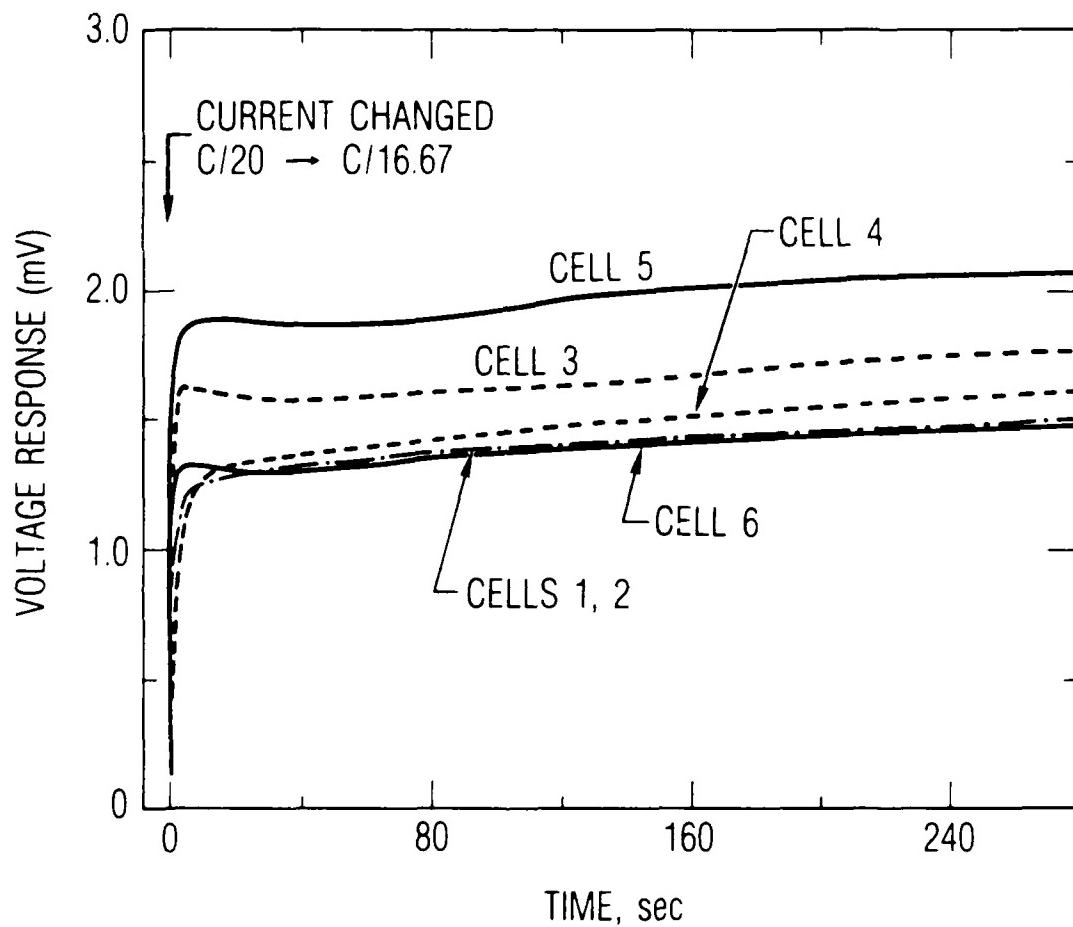


Fig. 2. NiCd "Activity" 8 hr into a C/20 Charge at 0°C. The "activity" is defined as the voltage response to a current step from C/20 up to C/16.67. Cell 6 is a 6 Ah cell that is considered normal; Cell 1 is a 50 Ah cell having very high overcharge voltages; Cell 2, a 50 Ah having slightly high overcharge voltages; Cell 4 had high internal pressures (10% more electrolyte than Cell 3); and Cells 3 and 5 had very high internal pressures during low temperature overcharge.

sensitive probe for tendencies to high pressure. In addition, the voltage behavior in Fig. 2 provides one of the few ways to probe individual electrode characteristics nondestructively within an operating NiCd cell.

III. CADMIUM ELECTRODE TESTS

Tests were done on a variety of cadmium electrodes from the cells previously described as well as several other electrodes from cells having both normal and atypical performance. These tests included stress cycling, electrochemical testing, and porosimetry analyses. All the electrochemical and stress cycling was done using 1 cm^2 electrode samples operated in an electrolyte flooded cell at room temperature using 31% KOH electrolyte. Mercury intrusion porosimetry was done using 1 cm^2 samples of the discharged electrode plates.

Stress testing was done by cycling cadmium electrode samples 10 times at a 10'/C charge and discharge rate. Active capacity was measured both before and after the stress cycling using C/5 charge and discharge rates, with residual capacity being evaluated at the C/100 discharge rate. The amount of material shed from the electrodes during the tests was determined by analysis for cadmium or nickel residues using atomic absorption analysis after dissolution of the residues in HNO_3 . The results from these tests did not allow the electrodes that were known to have weak sinter characteristics to be readily identified based either on the amount of material shed or on the changes in electrode utilization as a result of the high rate cycling. Little material was shed from any of the electrode samples, independent of whether they had poor sinter or a properly sintered porous substrate. In addition, no systematic correlations were found between cadmium utilization in the stress tests and the quality of the sinter. Electrochemical characterization of the cadmium electrodes was done using the test sequence outlined in Table 2.

The voltage characteristics during charge at C/10 for electrode samples with both good and poor sinter are indicated in Fig. 3. In Fig. 3 and in all of the data that were taken, only small differences in the behavior of the cadmium electrodes in the state of charge range in which the NiCd cell operates (indicated in Fig. 3) were found. Significant differences were, however, found in the overcharge, or hydrogen evolution region, for these electrodes. As indicated in Fig. 3 the electrodes having weak sinter always

Table 2. Test Sequence for Electrochemical
Characterization of Cadmium Electrodes

-
1. C/5 charge for 15 hr C rate corresponds to 20 mA/cm².
 2. C/2 discharge to depletion (defined as -0.5 volts vs. Hg/HgO).
 3. C/20 discharge to depletion.
 4. C/10 charge for 30 hr. Every 3 hr measure impedance and voltage at three rates: C/10, C/5, and C/2.
 5. Repeat steps 2 to 3.
 6. Cycle 10 times, each cycle consisting of a 2.5 hr charge at C/2 followed by discharge to depletion at C/2.
 7. Repeat steps 4 to 5.
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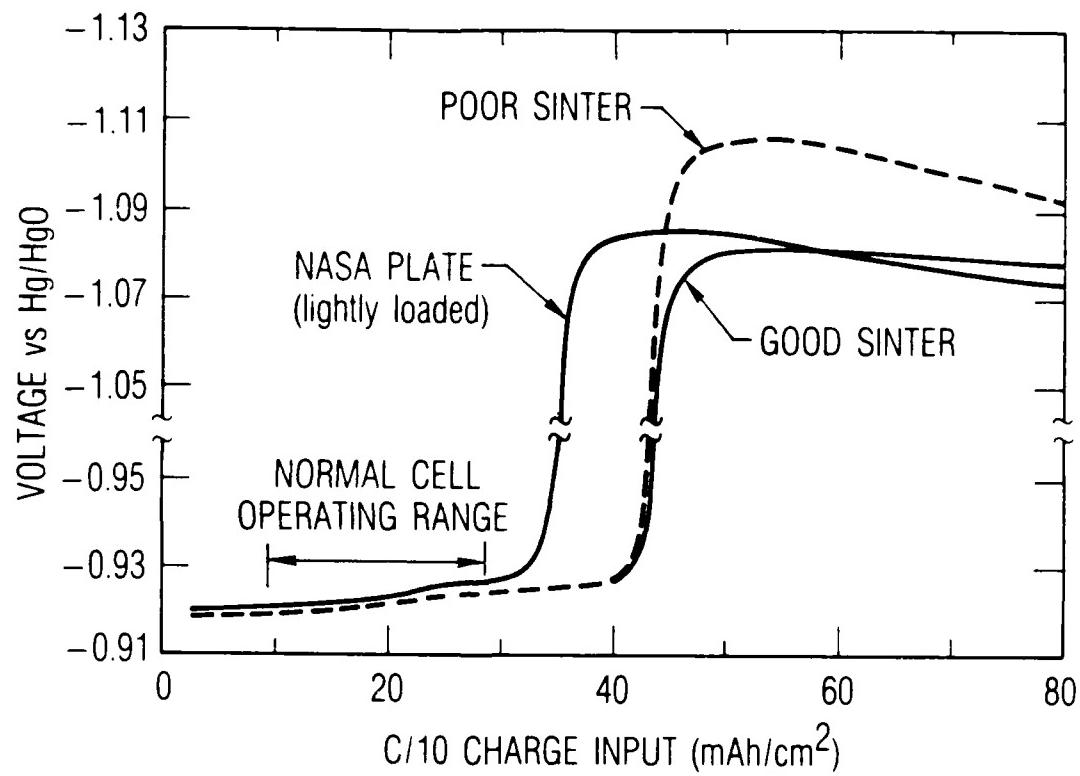


Fig. 3. Effects of Sinter Quality on the Overcharge Voltage of Cadmium Electrodes at 23°C.

had a higher hydrogen evolution voltage during step 7 in the sequence of Table 2 (prior to the 10 cycles in step 6 of Table 2 the electrode characteristics were not reproducible, depending on storage conditions). The higher hydrogen voltage was always accompanied by a higher electrode impedance during hydrogen evolution for the electrodes with weak sinter. The current/voltage behavior in the hydrogen evolution region indicated that the higher voltages for the electrodes with weak sinter were the result of a lower active surface area for gas evolution. In the case of one cadmium electrode that was tested, this correlation allowed detection of questionable sinter quality prior to detection by the cell user and cell manufacturer.

The behavior of cadmium electrodes from NiCd cells that exhibited excessive pressures during overcharge also was somewhat unusual in the hydrogen evolution region, as is indicated in Fig. 4. These electrodes exhibited low voltages and impedances for the hydrogen evolution reaction, with the voltage going through a significant peak. These tests did not clearly indicate why the hydrogen evolution reaction is accelerated, indicated by the decrease in overcharge voltage with time in Fig. 4 as hydrogen evolution continues from electrodes that were from cells having high overcharge pressures. This correlation may, however, be useful in detecting unfavorable operating conditions in cadmium electrodes.

Pore size distributions in cadmium electrodes are critical to maintaining sufficient electrochemical activity and to maintain appropriate gas channels for oxygen recombination. Porosimetry measurements were done on cadmium electrodes of all types that were included in this study, as well as on deloaded sinter from a number of the electrodes. These results indicated that three different pore distributions typically existed in the discharged cadmium electrodes. These distributions differ in the mean pore diameter associated with each, and are expected to result in significantly different distributions of active pores and electrolyte in the charged cadmium electrode. The different types of pore distributions are indicated in Fig. 5, along with a typical pore size distribution for the deloaded sinter. As indicated in the examples of Fig. 5 it is not unusual to have an overall pore size distribution that includes components from one, two, or three of the subdistributions. The

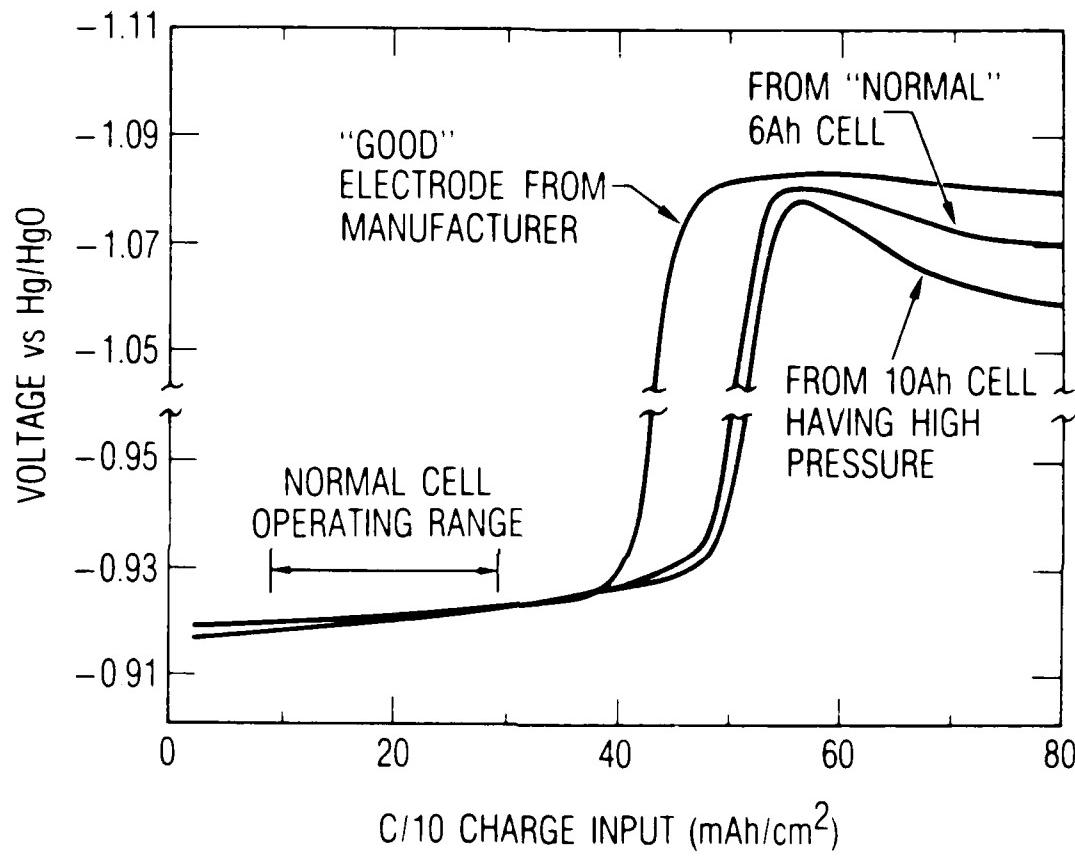


Fig. 4. Variation of Voltage with State of Charge for Cadmium Electrodes Using a C/10 ($2 \text{ mA}/\text{cm}^2$) Charge Rate at 23°C .

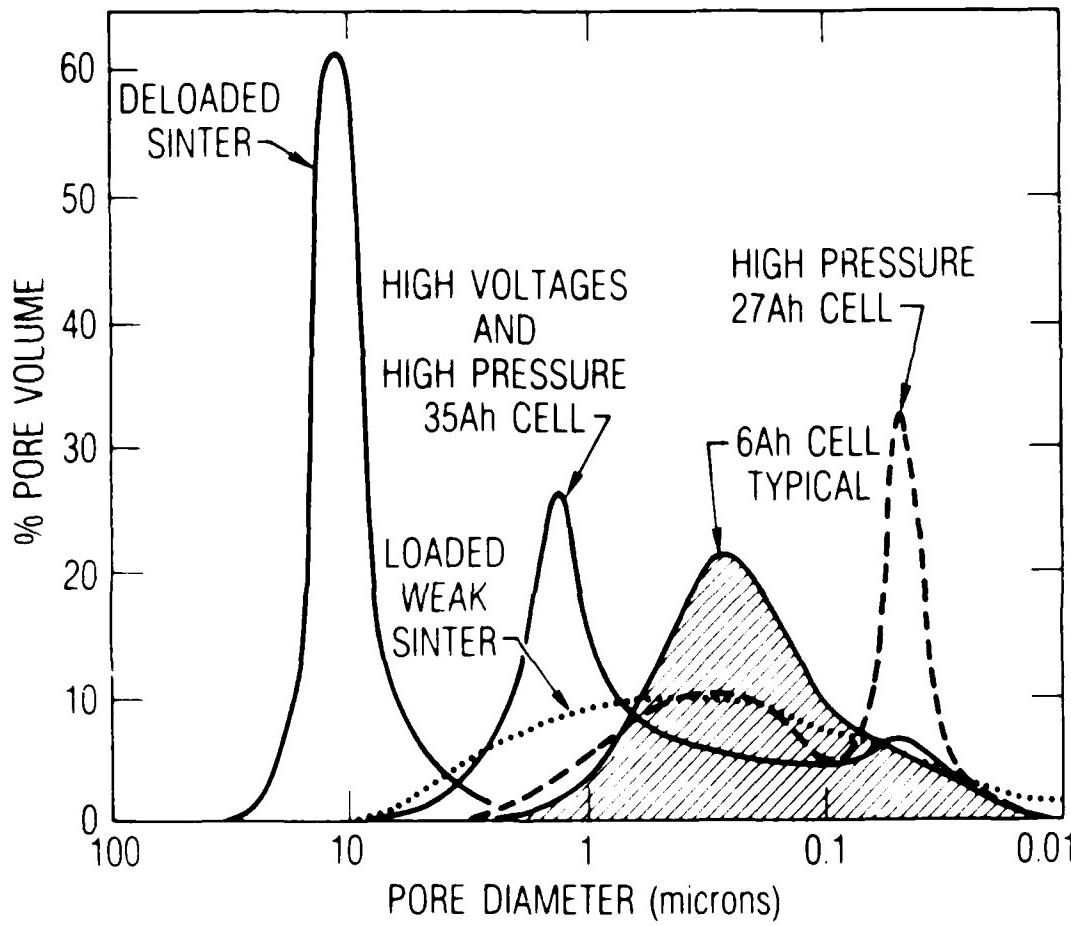


Fig. 5. Pore Size Distributions for Cadmium Electrodes Loaded with Active Material While in the Discharged State and for Deloaded Sinter.

three distinct subdistributions indicated in Fig. 5 peak in the pore size ranges of 1 to 2, 0.2 to 0.5, and 0.03 to 0.07 microns.

The pore size distributions indicated in Fig. 5 also had a close correlation to the performance of the cadmium electrodes in NiCd cells. Cells that exhibited high voltage and/or high pressures during overcharge tended to have a large fraction of the pore volume in either the large pore or the small pore distributions. Cells that operated in a normal fashion typically had a large fraction of the pore volume in the intermediate distribution centered between 0.1 and 1 micron. Furthermore, cadmium electrodes with weak or questionable sinter tended to have a significant fraction of pores larger than 1 micron, whereas electrodes from cells that only had high pressures tended to have more volume in the smaller pore distributions. The weak sinter was difficult to distinguish on the basis of the pore size distributions of the deloaded sinter, although careful analysis indicated a slightly broader distribution of pores in the weak sinter samples that were analyzed.

The correlation of the cadmium electrode pore distributions with cell performance characteristics indicated in Fig. 5 is actually reasonable based on fundamental considerations. The electrodes having a large amount of volume in very small pores develop high pressures during overcharge because the small pores will preferentially flood with electrolyte, and thus leave little cadmium surface area available to oxygen recombination. The weak sinter on the other hand appears to have internal voids that will inhibit effective electrochemical utilization of active material contained in these areas. The result will be a loss of active cadmium material and high voltages as the cadmium electrode is forced to operate closer to the fully charged state.

The results of the cell tests described here indicate a number of diagnostic tests that can be done at the cadmium electrode component level to detect incipient problems in cells containing these electrodes. The electrochemical tests can effectively detect either weak sinter or conditions that can cause high pressures during low temperature cell overcharge. The porosimetry measurements also provide an appealing technique for detecting conditions that could compromise cadmium electrode performance in the NiCd

cell environment. The drawbacks associated with the porosimetry in particular are the reproducibility and sample to sample variations that are present. For any analysis of electrode samples a significant number (at least 4-5) replicate analyses should be done to establish the statistical variability of the electrode properties being analyzed, as well as the range of "normal" characteristics expected. Another drawback of these methods for individual electrode characterization is that they require destructive analysis of at least one battery cell.

IV. CONCLUSIONS

The results that are reported here indicate that variations in cadmium electrode characteristics are responsible for the instances of high voltage and pressure during low temperature overcharge of NiCd cells that were provided for this study. While the amount of electrolyte in these cells can be varied to either raise or lower the overcharge pressure, this was found to be a factor that was secondary to the physical structure of the cadmium electrode active material deposit in cells with high pressures.

Tests that can be done on NiCd cells in a fully nondestructive way were shown to be capable of detecting conditions of weak sinter as well as potential high overcharge pressure conditions. Such tests could be easily applied on a screening basis to entire lots of flight NiCd cells, which would allow improved statistical correlations with performance to be established. Additionally, if cadmium electrode samples are available from disassembled cells, electrochemical and porosimetry tests have been demonstrated to provide an effective indication of conditions in cadmium electrodes that could compromise NiCd performance.

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